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IS 15854 (2009): Nuclear grade ino-exchange resins [CHD 30: Nuclear Materials]

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Indian Standard

NUCLEAR GRADE ION-EXCHANGE RESINS — SPECIFICATION

ICS 27.120.10

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 2009 **Price Group 4**

Nuclear Materials Sectional Committee, CHD 30

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Nuclear Materials Sectional Committee had been approved by the Chemical Division Council.

Nuclear grade ion-exchange resins are specially developed taking into consideration the problems associated with nuclear radiations and the necessity of continuous reuse of reactor waters over long period of time. They are chemically similar to non-nulcear grade resins but are characterized by high capacity in the desired ionic form, low fines, minimum leachable organic and very low levels of undesirable impurities. Resins with such stringent specification for use in nuclear reactors are designated as nuclear grade resins. Generally, they are used in systems where regeneration to the required form is not carried out due to system constraints.

The concept of water treatment in nuclear reactors differs greatly in view of safety and efficiency of operation. This arises mainly because of irradiation of impurities and corrosion products in the reactor core and consequent spread of radio activity at undesirable locations in the plant. In order to confine radiations to protected areas and reduce corrosion of all metallic and heat transfer surfaces, treatment of reactor waters using ion-exchange resins has been found to be convenient and economical.

The methods of sampling and test covered in IS 7330 : 1988 'Methods of sampling and test for ion-exchange resins (*first revision*)' are applicable to nuclear grade ion-exchange resins also. However, special test methods not covered in IS 7330 : 1988 have been included in this standard.

The composition of the Committee responsible for the formulation of this standard is given in Annex G.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
NUCLEAR GRADE ION-EXCHANGE RESINS — SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of test for nuclear grade ion-exchange resins, mainly used for purification of heavy water in pressurized heavy water reactor (PHWRs).

2 REFERENCE

The following standard contains provisions which, through reference in this text, constitutes provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
7330 : 1988	Methods of sampling and test for ion-exchange resins (*first revision*)

3 CLASSIFICATION

3.1 Cation-Exchange Resins

The material shall be of the following types:

a) Strong acid cation-exchange resins in H^+ form, and
b) Strong acid cation-exchange resins in Li^+ form.

3.2 Anion-Exchange Resins

The material shall be strong base anion exchanger (Type 1) in OH^- form.

4 REQUIREMENTS

4.1 The material shall be uniform, attrition resistant spherical beads and in moist swollen state.

4.1.1 *Cation-Exchange Resin*

The material shall be strongly acidic, cross-linked polystyrene matrix with sulphonic functional groups and exchange sites in hydrogen or lithium form. The material shall also comply with the requirements given in Table 1 when tested according to the methods referred in col 6 and 7 of Table 1.

4.1.2 *Anion-Exchange Resin*

The material shall be strongly basic, cross-linked polystyrene matrix with quaternary ammonium functional groups and exchange sites in OH^- form. The material shall also comply with the requirements given in Table 1 when tested according to the methods referred in col 6 and 7 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in moist condition in sealed air-tight plastic containers to avoid atmospheric contact and in quantities per pack as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 The containers shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net volume of the material;
d) Lot or batch number, in code or otherwise; and
e) Date of manufacture.

5.2.2 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

5.2.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Table 1 Requirements for Nuclear Grade Ion-Exchange Resins

(*Clauses* 4.1.1 *and* 4.1.2)

Sl No.	Characteristic	Requirement			Method of Test, Ref to	
		Cation Exchange Resin		Anion Exchange Resin OH^- Form	Cl No. in IS 7330	Annex
		Li^+ Form	H^+ Form			
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Description	Uniform, spherical beads supplied in Li^+ form in a moist swollen state	Uniform spherical beads supplied in H^+ form in a moist swollen state	Uniform, spherical beads supplied in OH^- form in moist swollen state	—	—
ii)	Particle size, mm (a minimum of 95 percent in between)	0.3 to 1.2	0.3 to 1.2	0.3 to 1.2	4.2.2	—
iii)	Effective size, mm	0.45 to 0.65	0.45 to 0.65	0.4 to 0.5	4.2.2.3	—
iv)	Uniformity coefficient, *Max*	1.8	1.8	1.8	4.2.2.4	—
v)	Fines content passing through 300 micron sieve, percent by volume, *Max*	0.5	0.5	0.5		A
vi)	Exchange capacity meq/ml, *Min*	1.8 in Li^+ form (a minimum of 98 percent of exchange sites should be in Li^+ form)	1.8 in H^+ form (a minimum of 98 percent of exchange sites should be in H^+ form)	0.8 in OH^- form (a minimum of 80 percent of exchange sites should be in OH^- form)	—	B
vii)	Chloride, *Max*	—	—	Less than 2 percent of exchange site	B-2.2	—
viii)	Moisture content, percent by mass, *Max*	55	55	60	4.7	—
ix)	Impurities, mg/l, *Max*:					
	a) Iron	80	80	80	—	C
	b) Copper	40	40	40	—	D
	c) Heavy metal as (Pb)	40	40	40	—	E
x)	Water soluble organic, mg $KMnO_4$/ml resin as supplied, *Max*	0.1	0.1	0.1	—	F

ANNEX A

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF FINES CONTENT

A-1 PROCEDURE

Place 100 ml of the resin as supplied on 300 micron sieve and agitate the sieve in vertical and horizontal planes while keeping the sieve under demineralized or distilled water. Do it for 10 to 15 min and collect the undersize fraction that has passed through the sieve. Measure its volume and express as fines content in percent by volume.

ANNEX B

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF EXCHANGE CAPACITY

B-1 DETERMINATION OF EXCHANGE CAPACITY OF NUCLEAR GRADE CATION EXCHANGE RESIN IN Li^+ FORM

B-1.1 Principle

Lithium capacity of the cation resin is determined by taking the difference between the total exchange capacity and the sum of the capacities due to impurity ions present in the resin.

B-1.2 Assumption

The most likely impurities on lithium form of nuclear grade cation resin are H^+ and Na^+.

B-1.3 Apparatus

B-1.3.1 *Column*, 15 mm diameter.

B-1.3.2 *Flame Photometer or Atomic Absorption Spectrophotometer (AAS)*

B-1.4 Reagents

B-1.4.1 *Hydrochloric Acid Solution*, 1 N (approx).

B-1.4.2 *Sodium Chloride Solution*, 5 percent.

B-1.4.3 *Sodium Hydroxide Solution*, 0.05 N (approx).

B-1.4.4 *Sodium Standard*, sodium chloride solution containing 100 µg Na/ml.

B-1.4.5 *Lithium Standard*, lithium sulphate solution containing 100 µg Li/ml.

B-1.4.6 *Screened Methyl Orange Indicator*

B-1.5 Procedure

B-1.5.1 Measure accurately 25 ml of tapped volume of the supplied resin as given in **4.2.2.1** of IS 7330 and transfer quantitatively into the column.

B-1.5.2 Wash the resin with DM water till no lithium is detected in the effluent (use flame photometer).

B-1.5.3 Pass about 500 ml of hydrochloric acid (1 N approx) at 8.3 ml/min through the column and collect the effluent in a 500-ml volumetric flask and make up to the mark.

B-1.5.4 Determine the sodium content of the elute in using suitable sodium standards and flame photometer or AAS. Express the results as '*A*' meq NA/ml of resin as supplied.

B-1.5.5 Determine the lithium content of the elute in **B-1.5.3** after proper dilution and using flame photometer with suitable lithium standards. This is to establish that the resin is mostly in lithium form.

B-1.5.6 Wash the resin free from hydrochloric acid using demineralized water. Pass about 500 ml of 5 percent sodium chloride solution through the column at a flow rate of 8.3 ml/min and collect the effluent in a 500-ml standard flask and make up to the mark. Titrate 25 ml of the elute against standard sodium hydroxide solution of normality 0.05 N using screened methyl orange indicator. Note the titre value as 'V_1' and calculate the total exchange capacity of the resin as '*B*' meq/ml of the resin.

B-1.5.7 Take another portion of the supplied resin as in **B-1.5.1** and repeat the step in **B-1.5.2**.

B-1.5.8 Pass about 500 ml of 5 percent sodium chloride solution through the column at a flow rate of 8.3 ml/min and collect the effluent in a 500-ml volumetric flask and make up to the mark. Titrate 25 ml of the elute against standard sodium hydroxide solution (0.05 N) using screened methyl orange indicator. Note the titre value as 'V_2'. Express the results as '*C*' meq H^+/ml of the resin.

B-1.6 Calculations

a) Total exchange capacity of the resin ('*B*') meq/ml $= 0.8 \times N \times V_1$

b) Exchange capacity due to H^+('*C*') meq/ml $= 0.8 \times N \times V_2$

c) Exchange capacity due to Na ('*A*'), meq/ml $= A$

d) Lithium capacity of the cation resin in meq/ml of resin $= B-(C+A)$

e) Li^+ capacity expressed as percent total exchange capacity $= \dfrac{B-(C+A)\times 100}{B}$

B-2 DETERMINATION OF EXCHANGE CAPACITY OF NUCLEAR GRADE CATION EXCHANGE RESIN IN H⁺ FORM

B-2.1 Apparatus

B-2.1.1 *Column*, 15 mm diameter.

B-2.1.2 *Flame Photometer or Atomic Absorption Spectrophotometer (AAS)*

B-2.2 Reagents

B-2.2.1 *Hydrochloric Acid Solution*, 1 N (approx).

B-2.2.2 *Sodium Chloride Solution*, 5 percent.

B-2.2.3 *Sodium Hydroxide Solution*, 0.05 N (approx).

B-2.2.4 *Sodium Standard*, Sodium chloride solution containing 100 µg Na/ml.

B-2.2.5 *Screened Methyl Orange Indicator Solution*

B-2.3 Procedure

B-2.3.1 Measure accurately 25 ml of tapped volume of the supplied resin as given in **4.2.2.1** of IS 7330 and transfer quantitatively into the column.

B-2.3.2 Rinse the resin with demineralized water till the effluent is free to methyl orange acidity.

B-2.3.3 Pass about 500 ml of 5 percent sodium chloride solution at the rate of 8.3 ml/min through the column and collect the effluent in a 500-ml volumetric flask and make up to the mark.

B-2.3.4 Titrate 25 ml of the elute from **B-2.3.3** against standard sodium hydroxide solution of normality, 0.05 N using screened methyl orange as indicator. Note the titre value as '*V*' ml and calculate the H^+ capacity of the resin as 'A' meq/ml of resin.

B-2.3.5 Take another portion of the supplied resin as in **B-2.3.1** and repeat the procedure as in **B-2.3.2**. Pass about 500 ml of 1 N (approx) hydrochloric acid solution at the rate of 8.3 ml/min through the column and collect the effluent in a 500-ml volumetric flask and make up to the mark.

B-2.3.6 Determine the sodium content of the elute in **B-2.3.5** using suitable sodium standards and flame photometer or AAS. Express the results as '*B*' meq Na^+ /ml of resin as supplied.

B-2.4 Calculations

a) Exchange capacity due to H^+ as ('*A*') meq/ml of resin $= 0.8 \times N \times V$

b) Exchange capacity due to sodium present in the resin as meq/ml of resin $= B$

c) Total exchange capacity (H^+ capacity + Na^+ capacity) $= A + B$

d) H^+ capacity expressed as percent of total exchange capacity $= \dfrac{A \times 100}{(A+B)}$

B-3 DETERMINATION OF EXCHANGE CAPACITY OF NUCLEAR GRADE ANION-EXCHANGE RESIN IN OH^- FORM AND CHLORIDE

B-3.1 Apparatus

B-3.1.1 *Column*, 15 mm diameter.

B-3.2 Reagents

B-3.2.1 *Sodium Nitrate Solution*, 4 percent.

B-3.2.2 *Standard Hydrochloric Acid Solution*, 0.05 N (approx).

B-3.2.3 *Standard Silver Nitrate Solution*, 0.05 N.

B-3.2.4 *Potassium Chromate Indicator Solution*

B-3.2.5 *Phenolphthalein Indicator Solution*

B-3.2.6 *Screened Methyl Orange Indicator Solution*

B-3.3 Procedure

B-3.3.1 Measure accurately 25 ml of tapped volume of the supplied resin as given in **4.2.2.1** of IS 7330 and transfer quantitatively into the column.

B-3.3.2 Rinse the resin with demineralized water till the effluent is free to phenolphthalein alkalinity.

B-3.3.3 Pass about 500-ml of 4 percent sodium nitrate solution at the rate of 8.3 ml/min through the column and collect the effluent in a 500-ml volumetric flask and make up to the mark.

B-3.3.4 Titrate 25 ml of the elute from **B-3.3.3** against standard hydrochloric acid solution using phenolphthalein indicator. Note the titre value as '*P*' ml.

B-3.3.5 Add two drops of screened methyl orange indicator and continue the titration till the colour changes to red. Note the titre value as '*M*' ml ('*M*' ml is the total volume of HCl of normality, 0.05 N consumed in both the titrations).

B-3.3.6 Pipette out another 25 ml aliquot of the solution from **B-3.3.3** and analyze for chloride as given in **B-2.2** of Appendix B of IS 7330. Note the titre value '*C*'.

B-3.4 Calculations

a) Exchange capacity in OH^- form as meq/ml of resin $= 0.8\,(2P - M)\,N$

b) Carbonate capacity as meq/ml of resin $= 1.6\,(M - P)\,N$

c) Chloride capacity as meq/ml of resin $= 0.8 \times C \times N$

d) Percent OH^- exchange sites $= \dfrac{(2P - M) \times 100}{M + C}$

e) Percent Cl^- exchange sites $= \dfrac{100 \times C}{M + C}$

ANNEX C

[*Table* 1, *Sl No.* (ix)(a)]

METHOD FOR DETERMINATION OF IRON IN ION-EXCHANGE RESIN

C-1 APPARATUS

C-1.1 Column, 15 mm diameter.

C-1.2 Spectrophotometer, visible.

C-1.3 *p***H Meter**

C-2 REAGENTS

C-2.1 Hydroxylamine Hydrochloride Solution, 10 percent.

C-2.2 Sodium Acetate Solution, 20 percent.

C-2.3 Orthophenanthroline Solution, 0.3 percent.

C-2.4 Concentrated Hydrochloric Acid

C-2.5 Iron Standard, ferrous ammonium sulphate solution containing 100 µg Fe/ml.

C-2.6 Hydrochloric Acid Solution, 1 N (approx).

C-2.7 Sodium Hydroxide Solution, 1 N (approx).

C-3 PROCEDURE

C-3.1 Place about 25 ml of the resin in the column and measure the volume accurately as in **5.1.3.1** of IS 7330.

C-3.2 Pass 500 ml of hydrochloric acid solution (approx 1 N) at the rate of 10 ml/min through the column.

C-3.3 Take 25 ml of the elute in **C-3.2** in a beaker and neutralize with sodium hydroxide solution (approx 1 N) to a *p*H of about 3 (use *p*H meter).

C-3.4 Add 1.0 ml hydroxylamine hydrochloride solution.

C-3.5 Add 3 ml of sodium acetate solution and 1.0 ml of orthophenanthroline solution.

C-3.6 Transfer the solution to 50-ml volumetric flask and make up the volume with DM water.

C-3.7 Mix well and allow to stand for 15 min. Measure the optical density of the solution on the spectrophotometer in a 1 cm cell at 512 nm.

C-3.8 Run a reagent blank along with the sample and measure its optical density.

C-3.9 Also run iron standards and obtain their optical density values.

C-3.10 Prepare a calibration curve using the corrected optical density values of the iron standards.

C-3.11 Read the iron content of the sample as '*A*' in µg using the corrected optical density of the sample with respect to blank:

$$\text{Iron } (\mu\text{g/ml of resin}) = \frac{20A}{25}$$

ANNEX D

[*Table* 1, *Sl No.* (ix)(b)]

METHOD OF DETERMINATION OF COPPER IN ION-EXCHANGE RESIN

D-1 APPARATUS

D-1.1 Column, 15 mm diameter.

D-1.2 Spectrophotometer, visible.

D-1.3 *p*H Meter

D-2 REAGENTS

D-2.1 Hydroxylamine Hydrochloride Solution, 10 percent.

D-2.2 Sodium Citrate Solution, 30 percent.

D-2.3 Sodium Acetate Solution, 20 percent.

D-2.4 Neocuproine Solution in Alcohol, 1 percent.

D-2.5 Concentrated Hydrochloric Acid

D-2.6 *iso*-Amyl Alcohol

D-2.7 Copper Standard (100 µg Cu/ml)

D-2.8 Hydrochloric Acid Solution, approx 1 N.

D-2.9 Sodium Hydroxide Solution, approx 1 N.

D-3 PROCEDURE

D-3.1 Place about 25 ml of the resin in the column and measure the volume accurately as in **5.1.3.1** of IS 7330.

D-3.2 Pass 500 ml of hydrochloric acid solution (approx 1 N) at the rate of 10 ml/min through the column.

D-3.3 Take 25 ml of the elute in **D-3.2** in a beaker and neutralize with sodium hydroxide solution (approx 1 N) using a *p*H meter.

D-3.4 Transfer the solution in **D-3.3** into a separating funnel and add 0.2 ml of concentrated hydrochloric acid. Add 1 ml of hydroxylamine hydrochloride solution and 10 ml of sodium citrate solution followed by 3 ml of sodium acetate solution. Add 1 ml of neocuproine solution. Extract the colour with 20 ml of isoamyl alcohol. Stopper and shake the separating funnel for about 2 min.

D-3.5 Allow to stand for 5 min for separation of phases and transfer the amyl alcohol phase into 1 cm absorption cell.

D-3.6 Measure its optical density on the spectrophotometer at a wavelength of 457 nm.

D-3.7 Run a reagent blank along with the sample and measure its optical density.

D-3.8 Also, run copper standards and obtain their optical density values.

D-3.9 Prepare a calibration curve using the corrected optical density values of the copper standards.

D-3.10 Read the copper content of the sample as '*A*' in µg using the corrected optical density of the sample with respect to blank:

$$\text{Copper } (\mu\text{g/ml of resin}) = \frac{20A}{25}$$

ANNEX E

[*Table* 1, *Sl No.* (ix)(c)]

METHOD OF DETERMINATION OF HEAVY METALS

E-1 APPARATUS

E-1.1 Column, 15 mm diameter.

E-1.2 Spectrophotometer, visible.

E-1.3 *p*H Meter

E-1.4 Nessler Cylinders, 50 ml capacity and Nessleriser.

E-2 REAGENTS

E-2.1 Hydrochloric Acid Solution, approx 1 N.

E-2.2 Sodium Hydroxide Solution, approx 1 N.

E-2.3 Hydrogen Sulphide Solution, saturated and freshly prepared.

E-2.4 Standard Lead Solution

Dissolve 0.183 1 g of lead acetate [$Pb(C_2H_3O_2)_2 \cdot 3H_2O$] in 100 ml of water and clear any cloudiness that might appear with a few drops of acetic acid. Dilute the solution to 1 000 ml. Take 100 ml of this solution and make it up to 1 000 ml. One millilitre of this solution contains 0.01 mg of lead (as Pb).

E-3 PROCEDURE

E-3.1 Place about 25 ml of the resin in the column and measure the volume accurately as in **5.1.3.1** of IS 7330.

E-3.2 Pass 500 ml of hydrochloric acid solution (approx 1 N) at the rate of 10 ml/min through the column.

E-3.3 Take 25 ml of the elute in **E-3.2** in a beaker and neutralize with sodium hydroxide solution (approx 1 N) using a *p*H meter. Add 0.5 ml of diluted hydrochloric acid and heat to 80°C. Immediately transfer to a Nessler cylinder and add 10 ml of hydrogen sulphide solution. Make up the solution to 50 ml. Carry out a control test using 2 ml of standard lead solution and the same quantity of reagents in the same total volume of the reaction mixture. Compare the colour of the solutions after 10 min. Intensity of the colour produced in the test solution shall be either equal or less as compared to that in the control test solution.

ANNEX F

[*Table* 1, *Sl No.* (x)]

DETERMINATION OF WATER SOLUBLE ORGANICS PRESENT IN NUCLEAR GRADE ION-EXCHANGE RESINS

F-1 PRINCIPLE

F-1.1 Most organic substances are destroyed by a boiling solution of acidified potassium permanganate. Water sample containing organics is refluxed with accurately measured amounts of sulphuric acid and potassium permanganate and the excess permanganate is titrated against standard oxalic acid. The amount of organic material in the sample is proportional to the potassium permanganate consumed.

F-1.1.1 *Assumption*

Since the method is based on oxidizing property of potassium permanganate, organic leachable removed from the resin is not expected to contain other oxidizable substances such as nitrites, ferrous iron, etc.

F-2 APPARATUS

F-2.1 Reagent Bottle with Ground Glass Stopper, 100 ml.

F-2.2 Round Bottomed Distilled Flask, with standard ground glass joint and 250-ml capacity.

F-2.3 Condenser, to match the joint in **F-2.2**.

F-3 REAGENTS

F-3.1 Potassium Permanganate, 0.01 N.

F-3.2 Sulphuric Acid, 9 N.

F-3.3 Oxalic Acid, 0.01 N.

F-4 PROCEDURE

F-4.1 Measure accurately 25 ml of tapped volume of the supplied resin as given in **4.2.2.1** of IS 7330 in a measuring cylinder and transfer it into a reagent bottle. Measure 50 ml of double distilled water and transfer it into a reagent bottle containing the resin. Stopper the bottle. Similarly keep in another bottle 50 ml of the double distilled water and stopper it to serve as blank. Keep the bottles for 24 h.

F-4.2 Shake the bottle containing the resin and allow to settle the contents. Pipette out 25 ml of the supernatant liquid and filter through a wet filter paper to retain any suspended resin particles on the filter paper. Transfer the filtrate into the distilling flask.

F-4.3 Pipette 50 ml of 0.01 N potassium permanganate solution into the flask.

F-4.4 Pipette 5 ml of 9 N sulphuric acid into the flask.

F-4.5 Add boiling chips to the flask to prevent bumping.

F-4.6 Attach the flask to the condenser and reflux gently by heating for 1 h.

F-4.7 Cool the flask to about 80°C and disconnect the condenser.

F-4.8 Add 50 ml of 0.01 N oxalic acid to the flask. Swirl the flask gently until the pink colour disappears.

F-4.9 Titrate the excess oxalic acid with 0.01 N potassium permanganate solution. Note the titre value as '*A*' ml.

F-4.10 Take 25 ml of double distilled water from the other reagent bottle and repeat the procedure from **F-4.3** to **F-4.9**. Note the titre value of potassium permanganate as '*B*' ml.

F-5 CALCULATION

Organic leachable $KMnO_4$ mg/ml resin = $(A-B) \times$ 0.025 28.

ANNEX G

(Foreword)

COMMITTEE COMPOSITION

Nuclear Materials Sectional Committee, CHD 30

Organization	*Representative(s)*
Bhabha Atomic Research Centre, Mumbai	Dr V. Venugopal (***Chairman***)
Atomic Energy Regulatory Board, Mumbai	Shri S. P. Agarwal
Bhabha Atomic Research Centre (Environment Division), Mumbai	Dr (Smt) G. G. Pandit
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